Name:……

Date:……..

5. SEPARATION OF MIXTURES, PURIFICATION OF SOLIDS

Objectives

Introduction to basic chemical laboratory operations: grinding, dissolving, decanting, centrifuging, filtration, crystallization. Separation of binary mixtures based on the different solubility of components, purification of the water soluble component by crystallization.

Pre-lab Assignment

5.1. Decantation, Centrifuging, Filtration

Pre-lab Exercise

1. How can we induce crystallization from the solution of a compound?

2. What are inclusions in a crystal? How can their formation be avoided during crystallization?

Decantation, Centrifuging, Filtration

Date: .....

Experiment Outline

The instructors demonstrate the use of different separation techniques.

1. What is the purpose of centrifuging a sample?

2. What is the purpose of decantation, when can it be used?

3. List the different types of filtration techniques and their area of laboratory use as shown in the demonstration.

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4. Draw the picture of a vacuum filtration apparatus fitted with a Büchner funnel and name its parts!

5.2. Purification of benzoic acid sample contaminated with sodium chloride

Date: .....

Experiment Outline

Weigh about 2 g of contaminated benzoic acid on a standard laboratory balance and dissolve it in ca. 15 cm^3 1:1 acetone:water mixture. After dissolution, filter the residue out on folded filter paper and rinse the beaker with ca. 5 cm^3 1:1 acetone:water mixture. Add ca. 80 cm^3 cold distilled water to the filtrate. Filter the precipitated crystals using a Büchner-funnel and an aspirator and wash them with ice-cooled distilled water.

The **purity** of benzoic acid can be **tested** as follows: collect the washing water going through the funnel in a test tube. Acidify it with some dilute nitric acid and add two drops of reagent solution of silver nitrate. White precipitation (AgCl) indicates a large amount of contamination, whereas a slightly turbid solution indicates a small amount of contamination. The solid is free of chloride only if the solution remains clear in this test after the addition of silver nitrate. Dry the purified chloride-free sample in air and weigh it.

Measure the melting point of the purified sample and save the rest for further experiments. Store the purified benzoic acid in a closed container.

Weighing of the contaminated sample:

Contaminated sample: ..... g

Observations during the precipitation of benzoic acid:

Write down the ***chemical equation*** of the silver nitrate test for chloride ion.

Result of the silver nitrate test:

Weighing of purified benzoic acid:

Mass of benzoic acid obtained: ..... g

Mass of contamination: ..... g

The amount of ***contamination*** of the original sample of benzoic acid was ..... % (m/m).

6. WORKING WITH GASES

Objectives

Introduction to the methods used for preparing pure and dry gases in the laboratory. Synthesis of a simple chemical using the reaction of a gas.

Pre-lab Exercises

1. Write down the equations of the chemical reactions that can be used to prepare the following gases in a chemical laboratory.

O_2:

H_2:

CO_2:

SO_2:

HCl:

2. What properties are very important for chemicals used to purify and dry gases?

6.1. Generation of gases in the laboratory

Date: .....

Experiment Outline

The instructors demonstrate how to use laboratory gas generator devices.

1. Gases can be prepared in the laboratory using the following methods *(summarize the most important features of each method based on the demonstration of the instructors)*:

2. Draw the diagram of a simple gas generator device in the chemical laboratory

6.2. Generation of hydrogen in Kipp's apparatus

Pre-lab Assignment

Find some background information about the following topics in a general chemistry textbook and read it.

- physical and chemical properties of hydrogen

Introduction

Strong, non-oxidizing acids react to form hydrogen with every metal that has a negative standard electrode potential. Strong basis react to form hydrogen with every *amphoteric* metal that has a negative standard electrode potential.

Pre-lab Exercises

1. Characterize hydrogen briefly.

color, odor:

state at room temperature:

density:

reactivity:

2. Complete the following chemical equations:

$Zn + NaOH + H_2O = Na_2[Zn(OH)_4] + H_2$

$Al + NaOH + H_2O = Na[Al(OH)_4] + H_2$

3. How is hydrogen gas generated in the laboratory? Write the relevant chemical equation(s).

Generation of hydrogen in Kipp's apparatus

Date: .....

Experiment Outline

Hydrogen is most conveniently prepared in the laboratory using Kipp's gas generating apparatus by the reaction of zinc with 20 weight% hydrochloric acid or 25-30 weight% sulfuric acid. Granulated zinc must be used in the apparatus. When the zinc used is very pure, a few drops of copper(II) sulfate solution are added to the acid to catalyze the steady formation of hydrogen gas.

The generated hydrogen may be contaminated with hydrogen chloride gas, this can be removed by bubbling hydrogen through water. Moisture in the hydrogen is removed by bubbling it through concentrated sulfuric acid or using a drying tube filled with calcium chloride.

If the generated hydrogen is to be burned at the outlet, a **hydrogen explosion test** must be done first because of the hazards arising from the explosive nature of mixtures of hydrogen and air. A small bent glass tube is attached to the outlet first, and the open end of this glass tube is immersed into a beaker which contains water. A test tube is filled with water and turned upside down so that its open end remains under the surface of water. The hydrogen bubbles are collected into the test tube until water is totally replaced by the gas. The open end of the test tube is then placed into the flame of a Bunsen burner. If a **sharp, whistle-like noise** is heard, the **hydrogen generated still contains some air** and the noise is the consequence of the explosion-like combustion of hydrogen. If the gas begins to burn **without noise**, the **hydrogen is pure** enough to be used. The Bunsen burner used for the hydrogen explosion test should not be close to the hydrogen generating apparatus (it must be placed outside the chemical hood) because some hydrogen may escape into the air during the test and

this might cause the whole apparatus to explode. **The hydrogen generating apparatus must always be used under a hood.**

1. Chemical equation of the hydrogen-generating reaction:

2. Draw the diagram of Kipp's gas generating apparatus and mark where the granulated zinc and the acid are located.

3. When hydrochloric acid is used for hydrogen generation in practice, its concentration does not usually exceed 20 weight%. Similarly, 25-30 weight% sulfuric acid is the most concentrated that is used for hydrogen generation. Find the reason for this practice.

Observations during the hydrogen explosion test *(positive and negative tests)**:***

Important safety information:

4. A test tube must not be used for the hydrogen explosion test for a second time without careful cleaning. Find the reason for this rule.

5. Why does a first negative test have to be repeated even if no noise can be heard at all?

Other notes: